THE NIST INDUSTRIAL THERMOMETER CALIBRATION LABORATORY

C. Dawn Vaughn and Gregory F. Strouse

National Institute of Standards and Technology

ABSTRACT

The NIST Industrial Thermometer Calibration (ITC) Laboratory provides calibrations of industrial thermometers over the range from –196 °C to 550 °C. The different types of thermometers include liquid-in-glass (LiG), thermistors, thermocouples (<200 °C), and industrial platinum resistance thermometers (IPRTs). Calibrations are performed by both comparison with a standard platinum resistance thermometer (SPRT) calibrated on the International Temperature Scale of 1990 and by a limited number of fixed-points. The comparison baths are liquid nitrogen (–196 °C), alcohol (–100 °C to 1 °C), water (0.5 °C to 95 °C), oil (95 °C to 275 °C), and salt (275 °C to 550 °C). The fixed points used are the melting point of ice (0 °C), the triple point of water (0.01 °C), and the melting point of gallium (29.7646 °C). The temperature measurement system for the reference SPRT it is a commercially-available ac resistance ratio bridge, for the IPRTs and thermistors it is a dc voltmeter with a constant current source, for the thermocouples it is a dc voltmeter, and for the LiGs it is a digital video camera. All of these temperature measurement systems are integrated via three computer-controlled data acquisition systems that semi-automate calibrations. This paper presents the methods, equipment, and uncertainties associated with the calibration of industrial thermometers in the ITC Laboratory.

1. INTRODUCTION

The NIST Industrial Thermometer Calibration (ITC) Laboratory supplies calibrations, based on the International Temperature Scale of 1990 (ITS-90), of industrial thermometers over the temperature range from –196 °C to 550 °C to industry, government owned facilities, and secondary calibration laboratories. The types of thermometers calibrated in the ITC Laboratory are liquid-in-glass (LiG) thermometers, thermocouples [(TC), < 200 °C], thermistors, and industrial platinum resistance thermometers (IPRTs). Calibrations are performed by comparison with an ITS-90 calibrated standard platinum resistance thermometer (SPRT) and by the fixed points of the melting point of ice [MP, (0 °C)], the triple point of water [TPW, (0.01 °C)], and the melting point of gallium (27.7646 °C). Three computer-controlled data-acquisitions systems were developed to automate the calibration of these industrial thermometers over the range from –196 °C to 550 °C.

2. THERMOMETER CALIBRATIONS

The types of thermometers calibrated and the temperature ranges available for those calibrations are given in Table 1. Several different types of calibrations are available for customers. In order to provide maximum flexibility to the customer, the calibration temperatures and type of calibration report are chosen according to the intended use of the thermometer and the needs of the customer. Suggested calibration temperatures and types of calibration reports are given to customers on request.

The LiG thermometers are calibrated by comparison with an ITS-90 calibrated SPRT over the range from –196 °C to 400 °C and at the ice MP. The different types of LiG thermometers that are calibrated include Hg, organic, or proprietary non-toxic filled. There is a required minimum of two calibration temperatures per thermometer. The calibration report provides an ITS-90 based table of measured bath temperature versus LiG thermometer correction and calibration uncertainties.

Table 1. The types of calibrated thermometers and temperature ranges available for calibration in the NIST Industrial Thermometer Calibration Laboratory.

Thermometer Type	Temperature Range, °C		Thermometer Type	Temperature Range, °C
LiG (Hg), Partial/Total Immersion	0 to 400		IPRT	–196 to 550
LiG (organic), Partial/Total Immersion	–200 to 0		Digital	–196 to 550
ASTM*, Part/Total Immersion	–70 to 400		Thermistor	–50 to 100
Clinical	80 to 135		Thermocouple	–196 to 300
Calorimetric, Total Immersion	0 to 51			

*ASTM = American Society for Testing and Materials

IPRTs are calibrated by comparison with an ITS-90 calibrated SPRT over the range from –196 °C to 550°C. The calibration report provides an ITS-90 based table of measured bath or fixed-point temperature versus thermometer resistance, calibration uncertainties, and the thermometer calibration coefficients.

Thermistor thermometers are calibrated by comparison with an ITS-90 calibrated SPRT over the range from –50 °C to 100 °C. In addition, fixed-point calibrations are provided at the TPW, the ice MP, and the Ga MP (thermometer o.d. <3.6 mm). The calibration report provides an ITS-90 based table of measured bath or fixed-point temperature versus thermometer resistance, calibration uncertainties, and the thermometer calibration coefficients.

Thermocouples (TCs) are calibrated by comparison with an ITS-90 calibrated SPRT over the range from –196 °C to 300 °C. The calibration report provides an ITS-90 based table of measured bath temperature versus emf and uncertainties. Type T thermocouples require six calibration temperatures to produce a table of temperature, emf and *dV/dt* in one degree increments.

Digital Readout Thermometers consisting of a thermometer probe and a measurement readout device are calibrated as a unit by comparison with an ITS-90 calibrated SPRT over the range –196 °C to 550 °C. The calibration report provides an ITS-90 based table of measured bath temperature versus digital thermometer reading and calibration uncertainties. The uncertainty of the digital readout device is unknown and must be included by the user of the device.

Special calibrations of industrial thermometers are performed on request. Some of the thermometer types that require special calibrations are air probe, prototype, and autoclave thermometers.

3. CALIBRATION EQUIPMENT

The reference temperature measurement system consists of a commercially-available 9½ digit automatic ac resistance ratio bridge, a thermostated reference resistor (36 °C ±0.01 °C), and three ITS-90 calibrated SPRTs. The reference SPRTs are calibrated by ITS-90 fixed-points in the NIST PRT Calibration Laboratory over the range from –189.3442 °C (Ar TP) to 660.323 °C (Al FP). The SPRTs are measured at the TPW after each use in a comparison bath to verify their calibration status. If a reference SPRT changes by more than the equivalent of 0.001 °C at the TPW, then the PRT Laboratory recalibrates the SPRT. The operational capability of the ac resistance ratio bridge is verified yearly and the reference resistor is calibrated yearly.

The calibration system for LiG thermometers uses a new computer-controlled, digital video camera system consisting of a digital video camera, a 10X macro lens, a computer-integrated marking and measurement grid controller, and a high-resolution monitor. The camera is mounted on a tripod to provide stability (pitch and yaw) and for easy mobility to move the measurement system from bath to bath. The resolution of the digital video system is 1/34 of the scale division of the LiG thermometer.

The calibration system for thermocouples consists of an 8½ digit voltmeter, a 20 channel low thermal scanner, a low-thermal connection box, an ice MP reference-junction bath, and a 10 mV reference source. The 8½ digit voltmeter is calibrated yearly using a NIST calibrated 10 V reference source.

The calibration system of the IPRTs and thermistor thermometers is a dc measurement system consisting of an 8½ digit voltmeter, a 100 Ω dc resistor (IPRTs), a 10 kΩ dc resistor (thermistors), a constant current source, and a scanner. The IPRTs, thermistor thermometers, and resistors are measured as four-terminal devices. The excitation current supplied in series to the current leads of the IPRTs and 100 Ω dc resistor is 1 mA; and either 1 µA and 10 µA for the thermistors and 10 kΩ dc resistor. The 8½ digit voltmeter is calibrated yearly using a NIST calibrated 10 V reference source. The dc reference resistors are calibrated yearly.

The comparison baths, range of temperature, number of thermometers and performance parameters of the baths are given in Table 2. The comparison baths use an integrated thermometer holder that allows for the rotation of the thermometers to the same measurement position as that of the reference SPRT.

Table 2: The type of baths, range of temperature of each bath, number of thermometers that can be placed in the bath, and uncertainty component of each bath used in comparison calibrations.

Bath type	**Temperature range °C**	**Number of Thermometers**	**Maximum Instability ±m°C**	**Maximum Non-uniformity ±m°C**
Cryostat	–97 to 1	8	5.0	6.2
Water	0.5 to 95	23	1.5	0.8
Oil	95 to 275	23	3	2.5
Salt	275 to 550	11	4	5

The cryostat is a commercially built bath used over the range from –97 °C to 1 °C. Ethanol is used as the bath medium. The test well of the bath is approximately 41 cm deep and 10 cm in diameter. The section for thermometers is isolated from the heating and cooling coils using a weir to control the flow of the stirred bath medium. A special view port allows for the measurement of the meniscus of total immersion LiGs at the liquid surface layer.

The water bath is a commercially built bath used over the range from 0.5 °C to 95 °C. Distilled water is used as the bath medium. This test well of the bath is approximately 61 cm deep and 25 cm by 30 cm. The stirring mechanism, heating coils, and cooling coils are isolated from the section holding the thermometers.

The oil bath is a commercially built bath used over the range from 95 °C to 275 °C. A high-temperature silicone oil is used as the bath medium. The test well of the bath is approximately 61 cm deep and 19 cm in diameter. The section for thermometers is isolated from the heating coils using a weir to control the flow of the stirred oil.

The salt bath is a commercially built bath used at temperatures over the range from 275 °C to 550 °C. The bath medium is a salt consisting of potassium nitrate (53%), sodium nitrite, (40%), and sodium nitrate (7%). The test well of the bath is approximately 43 cm deep and 10 cm in diameter. The section for thermometers is isolated from the heating coils using a weir to control the flow of the stirred salt.

The ice melting point is realized using shaved distilled-water ice and distilled water compacted into a Dewar flask [1]. The amount of liquid water added to the Dewar fills the voids between the shaved ice particles, but not enough to float the ice. A siphon hose is inserted to the bottom of the Dewar to drain off the excess water as needed. After an equilibration time of 30 min elapses, the excess water is drained and more ice is added to the top of the Dewar. A clean rod of suitable diameter is inserted into the ice to make a slip-fit hole for the thermometer. The thermometer is allowed to equilibrate for 10 min before measurement. Gloves are worn during the fabrication of the ice point to minimize the possibility of contamination of the water. An opaque cover is placed over the top of the Dewar to eliminate incident radiation.

The TPW is realized using two commercially fabricated Type A TPW cells with thermometer well inner diameters of 11 mm and 13 mm. The TPW cells are maintained in a commercially available Peltier-cooled bath that accommodates up to four cells. The stability of the water filled, air-stirred maintenance bath is ±2 m°C and can maintain a TPW mantle for at least four weeks.

The Standard Reference Material® (SRM®) 1968 Ga MP cell can be used to measure thermometers with outer diameters not exceeding 3.6 mm [2]. A thermal enclosure is used to realize the Ga MP with a plateau duration of about four h.

4. COMPUTER DATA ACQUISITION PROGRAMS

The computer data-acquisition system consists of three programs for measuring LiG thermometers, IPRT and thermistor thermometers, and thermocouple thermometers. The programs were written using an object-oriented software package. All three of the programs save two files with one containing all of the measured data and the second containing a summary of the measured temperature and the measured value or temperature correction of the thermometers under test. Multiple copies or combinations of the three programs can be used simultaneously to increase the flexibility of the thermometer types that are being calibrated.

In all of the three programs, a calibration matrix screen allows the user to change the settings of the 9½ digit automatic ac resistance ratio bridge, enter the serial number of the reference SPRT, the serial number and channel number of each test thermometer, the serial number and channel number of the check thermometer, and the calibration temperatures to be measured. Additionally input information includes: the measurement excitation current(s) for the IPRT and thermistors; and the thermometer graduation, temperature scale (°C or °F), and stem temperature correction requirement for the LiG thermometers. From the matrix, the program creates a measurement macro that groups thermometers at the same temperature in blocks of four (three test and one check thermometer). The read order for a measurement block is the reference SPRT, the reference resistor, the test thermometers, the check standard, the SPRT; then in reverse order, the check standard, the test thermometers, the reference resistor (reversed current) and then a final SPRT reading. The read order is reversed to counteract any drift that may occur during measurements. When fixed points are used, the program will skip the reading of the reference thermometer. On completion of the measurement block for a given temperature, the program computes the drift in temperature of the comparison bath from the three reference SPRT readings. If the drift is within acceptable limits, either the next measurement block is measured (if necessary) or the bath is set for the next temperature. When all required measurements are completed, the SPRT is measured at the TPW and the program calculates the final comparison temperatures. The check SPRT is used as total systems check of the comparison measurements.

Comparing the results from the three programs with the results calculated by both hand and electronic spreadsheet using the raw measurements performed the validation step of the three programs. The three methods agree to within 0.01 m°C. A detailed description and validation process of the three programs with pictures of the computer screens is found in reference 3.

5. CALIBRATION OF THERMOMETERS

As a check on the stability of a thermometer during calibration, the thermometer is always measured at either the ice point or the TPW prior to calibration and then again at the end of calibration. If requested, these values are reported to the customer, otherwise the information is used only to study the stability of industrial thermometers. For IPRTs and thermistors, some customers require additional data points that are not used in the model used in fitting the data. These extra measurements are used to check the uncertainty in both the model and in the calibration of the thermometer.

A thermometer design with which NIST personnel are unfamiliar will undergo a check of its immersion characteristics in each applicable bath to validate proper immersion of the thermometer. Depending on the construction the thermometer (sheathed or bare element), the bath liquid, the calibration temperatures required, and the intended use of the thermometer (air or liquid); the thermometer will be immersed either directly in the bath liquid, in a closed-end glass tube containing air, or in a closed-end glass tube containing a non-electrically, high-thermally conductive fluid. The sensor of the reference SPRT is placed in the same horizontal plane of the bath as that of the thermometer sensor to minimize effects of any vertical thermal gradients.

LiG are inspected for acceptance by checking under a 10X microscope for defects, which may include foreign material in the capillary, improperly numbered graduations, or non-uniform markings [4]. The first measurement is at the ice point, followed by measurements in ascending order of temperature, and following measurements above room temperature, the thermometers are finally retested at the ice point after a 72 h rest period. Fadens or auxiliary thermometers are used for stem temperature corrections, which are applied to American Society for Testing and Materials (ASTM) thermometers [5].

IPRTs and thermistors are inspected to determine the appropriate reference resistor and current excitation. For the excitation current, IPRTs are tested at 1 mA, while thermistors are tested at 1 µA or 10 µA. The IPRTs and thermistors are either measured at the ice MP or TPW, then they are measured in ascending temperature, and finally are remeasured at either the ice MP or the TPW.

Thermocouple junctions are fabricated if required by silver soldering the wires together. Fiberglass sleeving is used to insulate the wires if it is a bare wire thermocouple. The test TC is placed in a glass tube before insertion into the calibration baths. The TC wires are connected to two copper extension wires from the same lot of wire and these junctions are maintained at an ice point, which is the reference junction. The measurements are taken in ascending temperature

Digital thermometers are entered in the calibration system under the LiG program. Digitals can be a variety of types of thermometers (e.g. IPRT, TC, or thermistor). The digitals are either measured at the ice point or the TPW, then measured in ascending temperature and finally the ice point or TPW.

6. UNCERTAINTIES

The estimated uncertainty assigned to thermometer calibration is derived using uncertainty components from the reference temperature measurement system, the industrial thermometer measurement system, the comparison baths, the fixed-point cells, and the thermometer type (e.g. graduation of LiG thermometer). The uncertainty derived from the reference temperature measurement system, comparison baths and fixed-point cells is applicable for each type of industrial thermometer calibrated. The uncertainty derived from the industrial thermometer measurement system is unique for each type of industrial thermometer calibrated. As an example, the uncertainties assigned to the comparison baths and fixed points are given in Table 3. The U (k=2) assigned to the thermometer calibrations for the given temperature range are given in Table 4. Detailed descriptions of the uncertainty components and assigned uncertainties are found in references 3, 6-8.

Table 3: U (k=2) assigned to the NIST ITC Laboratory comparison baths and fixed-points.

Comparison Bath	**Cryostat**			**Water**	**Oil**	**Salt**
Temperature range, °C	–97 to –80	–80 to –70	–70 to 1	0.5 to 95	95 to 300	275 to 550
***U* (*k*=2), m°C**	9	4	2.3	2.4	4.8	7.5

Fixed point	**Ice MP (0 °C)**	**TPW (0.01 °C)**	**Ga MP (29.7646 °C)**
***U* (*k*=2), m°C**	1.8	1.4	1.6

Table 4: U (k=2) assigned to the NIST ITC Laboratory thermometer calibrations.

Thermometer Type	***t* range °C**	**Max *U* (*k*=2)[†] °C**		**Thermometer Type**	***t* range °C**	**Max *U* (*k*=2) °C**
LiG Hg part.	0 to 500	≤ 1.0		IPRT	–196 to 550	0.009
LiG Hg total	0 to 500	≤ 1.0		Thermistor	–50 to 100	0.003
LiG org. part.	–90 to 0	≤ 1.0		Digital[*]	–196 to 550	0.009
LiG org. total	–200 to 0	≤ 0.5		TC type T	–196 to 300	0.9
Calorimetric	0 to 51	≤ 0.01				

[*]Digital thermometer uncertainty does not include the unknown uncertainty of the readout device.
[†]Assigned uncertainty depends on thermometer graduation of LiG [6].

7. CONCLUSION

The addition of the computer-controlled data acquisition systems and the digital video camera calibration system in the ITC Laboratory at NIST has improved the efficiency, accuracy and usability in calibrating industrial thermometers.

8. REFERENCES

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Address of the Authors:

C. Dawn Vaughn and Gregory F. Strouse, NIST, 100 Bureau Dr., MS 8363, Gaithersburg, MD 20899 USA, E-mail: cvaughn@nist.gov and gstrouse@nist.gov, http://www.cstl.nist.gov/div836/836.05/home.htm